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Raman / Bench Scale Polymerization of Ethylene

Purpose: To determine whether or not a Raman filter is needed on a simulated reactor containing polymer and if so, to determine whether or not the filter will provide an acceptable spectrum.

See 36556-25, J.B. Ashew

Time	File	
11:50	Slurry 1	30 sec acquisition, no filter, IC4 + PE fluff
	Slurry 2	60 sec " " " " "
	Slurry 3	30 sec acquisition, no filter, " " "
11:15	Slurry Filter 30 sec	" " " with filter 60°C, 112 psig
	SLF0001	60 sec acquisition
11:25	SLF0001	60 sec, 60°C, 112 psig, 700 rpm, Ti + SST support polyethylene
	SLF0002	repeat of 1
	SLF0003	Go to 73°C
	SLF0004	
	SLF0005	73°C, 124 psig
	6	80°C, 130 psig
	7	" "
	8	Transition/heat up to 90°C.
	9	90.2°C, 224 psig
	10	90.2°C, 224 psig
	11	Transition to 100°C
	12	100.7, 275 psig
	13	100.1, 273 psig
	14	104.4, 295 psig
	15	105.3, 305 psig
	16	105.1, 301 psig
	17	60.2, 113 psig
	18	Added ethylene to get total reactor pressure = 200 psig
	19	60°C, 190 psig (C ₂ band forward) OK/SRS 113 psig IC4 + 87 psig C ₂
	20	60°C, 198 psig
	21	Group to 73°C on this scan.
	22	75°C, 210 psig
	23	↓ ↓ ±4°C fluctuations
	24	
	25	76°C, 210 psig
	26	71.3°C, 240 psig

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Roman / Branch Scale Polymerizer at 700 RPM

Purpose: To Return more data using a Roman Polymerizer
 The Reactor contains 450 grams Polymer & 2 L of ICH
 + stirring at 700 RPM.

8:29 - started.

SLFA - Scan

2 L ICH Polymer 450 gram RPM 700

1	60.0	115	Psy	
2	60.0	115	Psy	
3	83.6	115	Psy	Rx Trying to control Temp.
4	80.0	183	Psy	OK
5	80.0	183	Psy	OK
6	102.5	299	Psy	Rx Trying to control Temp.
7	100.1	297	Psy	OK
8	100.0	297	Psy	OK
9	100.0	361	Psy	Add C ₂ H ₅ Br, Rx Trying to control Temp.
10	100.0	361	Psy	OK
11	100.0	362	Psy	OK
12	78.0	270	Psy	Rx Trying to control Temp.
13	85.0	273	Psy	Rx Trying to control Temp.
14	81.4	284	Psy	Need to add C ₂
15	80.2	270	Psy	OK
16	80.0	271	Psy	OK
17	64.0	-	-	Rx Trying to control Temp.
18	59.8	196	Psy	Rx Trying to control Temp.
19	60.0	186	Psy	Need C ₂
20	60.0	200	Psy	Rx Trying to control Temp.
21	60.0	197	Psy	OK
22	60.0	197	Psy	Done.

500 C₂H₅Br added for 878Scan 1 thru 22 C₂

was maintained set Psy

for 860C, 80C, 100C

115 Psy, 183 Psy, 299 Psy

+ 878 Psy, 878 Psy, 878 Psy

202 Psy, 270 Psy, 364 Psy

Change to Gas Phase Probe

Scan 23	out side light (Room)	Gas Spectrum
24	59.7	186 Psy
25	60.0	182 Psy

Reactor Filter on - Change to Liquid Phase Probe

Change C₂H₅Br to 250 Torr. Rx by at 60C, stirring 700 RPM

10:48

SLFB - Scan

1	60.0	245	Psy	
2	60.0	251	Psy	OK
3	60.0	245	Psy	OK
4	82.0	383	Psy	Rx Trying to control Temp.
5	77.0	287	Psy	" "
6	84.0	340	Psy	" "
7	78.0	329	Psy	
8	94.0	422	Psy	Rx Trying to control Temp.
9	100.0	400	Psy	" "
10	103.0	410	Psy	" "
11	91.0	377	Psy	Temp Cycle
12	103	512	Psy	" "

Stop for hours

Cooling Reactor

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Continued From Page 33 325-34				stand by back from lunch.	
P. 1	SLFBZ 0001	- 66.0°	338	Psy	Add 2° P ₂ to set 328 P ₂
IC4	Scan-2	- 64.0°	355	Psy	Temp not control, Real good updr.
	2	- 57.0°	269	Psy	OK.
	4	- 55.7°	283	Psy	Raise Temp to 60°
RAM 700	5	- 87.0°	98.0	Psy	
	6	- 77.0°	333	Psy	
	7	- 60.0°	444	Psy	
	8	- 83.1°	358	Psy	Psy is up + Down?
	9	- 85.8°	450	Psy	Raise Temp to 100°
1 Temp	10	- 100.0°	569	Psy	
	11	- 42.0°	425	Psy	Temp of Rx was 104°
2 Temp	12	- 104.0°	569	Psy	Up + Down Temp.
	13	- 45.0°	445	Psy	" " "
	14	- 96.0°	517	Psy	" " "
	15	- 105.0°	475	Psy	
3 Temp	16	- 72.0°	288	Psy	Cooling to 60° →
	17	- 58.0°	305	Psy	
	18	- 62.0°	299	Psy	
4 Temp	19	- 55.0°	236	Psy	25 grams H ₂ O added
5 Temp	20	- 64.0°	280	Psy	
	21	- 72.0°	332	Psy	Temp up to 80°
	22	- 83.0°	279	Psy	50 grams of H ₂ O added
Temp	stop to			Flare Rx	NOTE: (To open to Inspect Probe)
1 Temp	SLFBZ 000	- 1	3.7°	214	Psy
2 Temp	"	2	0.1	0	Psy
3 Temp	"	3	0.4	0	Psy
4 Temp	SLFBZ 000	-			Flare Rx for 20 min Pressured up with N ₂ to 200 P ₂
5 Temp	Scan 21	30.0°	53	Psy	Stirring on, Polymer, vapors
	2	30.0°	83	Psy	NO Pressure open to Flare
	3	30.0°	72	Psy	Stirrer Stop
	4	30.0°	72	Psy	Purge Rx with N ₂
	5	30.0°	72	Psy	N ₂ Flow Thru Rx
	6	30.0°	72	Psy	2) 2 IC4 + stirring 700 RPM
	7	30.0°	72	Psy	with Filter + no Polymer
	8	30.0°	72	Psy	70 grams H ₂ O added
	Stop Down Time to 80 min				Flare Rx over night
	Note: When I clean Rx out of Polymer the Polymer had stuck to the bottom of Rx + some of the wall. (the Probes were clean.)				